STATE OF MICHIGAN
DEPARTMENT OF
ENVIRONMENT, GREAT LAKES, AND ENERGY
LANSING

GRETCHEN WHITMER
GOVERNOR

LIESL EICHLER CLARK
DIRECTOR

August 20, 2020

VIA E-MAIL and U.S. MAIL

Mr. Jim Saric
Remedial Project Manager
United States Environmental Protection Agency
Region 5
77 West Jackson Boulevard (SR-6J)
Chicago, Illinois 60604-3511

Dear Mr. Jim Saric:

SUBJECT: Michigan Department of Environment, Great Lakes, and Energy (EGLE) comments on the Area 1 Pre-Design Investigation (PDI) Evaluation Report Part 2 – Near-TCRA Floodplain Soil (Report). Kalamazoo River Area 1, OU5 Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund site.

The Report provides a summary of PDI data that was collected from the floodplains located in the former Plainwell impoundment as part of the Remedial Design (RD) in Area 1 of Operable Unit 5 (OU5) of the Kalamazoo River Superfund Site (Site) consistent with requirements specified in the Unilateral Administrative Order for RD and Remedial Action (CERCLA Docket No. V-W-17-C-002). The Report summarizes the PDI data that was collected in 2017 and 2018 from the Plainwell floodplain and generally concludes that: no work is necessary to protect ecological receptors; two relatively small areas in the floodplain exceed the site-specific recreational criteria, some access roads appear to have been removed while others remain, and; some banks have failed and require repair, and those details will be outlined in a future RD submittal. A few key comments are briefly discussed below and detailed comments on the Report are provided as an attachment.

EGLE had hoped to avoid providing comments related to data quality concerns on this Report since EGLE believed this was a topic that had been well documented, and was being explored and resolved cooperatively by the Work Group. EGLE believes key details and information collected during the PDI regarding the nature and extent of contamination were omitted from the report. Specifically, the Report mentions that split samples were taken but does not split sample results, which suggest that the PDI data is biased low. EGLE's split samples had total polychlorinated biphenyl concentrations that were an average of 2.4-times greater than the parent sample analyzed by Georgia-Pacific's (GP's) lab. The Report also contains inappropriate and suggestive language in the narrative regarding the "superiority" of PDI data. Therefore, EGLE ultimately drafted several comments on the Report related to data quality. EGLE appreciates the United States Environmental Protection Agency's (U.S. EPA's)

willingness to open a dialogue among all Parties, the cooperative dialogue between the U.S. EPA Region 5 Chemists and EGLE throughout this process, and the significant effort they have undertaken to try to expeditiously resolve any data quality issues that may exist. EGLE seeks to work cooperatively with all Parties and believes that discussions that are currently underway will likely improve data quality.

EGLE appreciates the Respondents acknowledging that the banks in the former Plainwell impoundment that were excavated and restored as part of the Time Critical Removal Action have failed and require repair. However, EGLE notes that the timeline provided in the narrative is inconsistent with historical records and disagrees that the Bank Monitoring Reports have identified all areas that are unstable or have eroded. For example, bank failures were documented and repairs to the banks (i.e. addition of rock) were made prior to the Plainwell TCRA being declared complete, which was in 2012. Erosion has also been documented in other areas of the former Plainwell impoundment, including the right descending bank above and below the US-131 highway bridge. Following completion of the Plainwell TCRA, EGLE evaluated the channel morphology by comparing the characteristics in the restored reach of the former Plainwell impoundment to local, undisturbed reference reaches on the Kalamazoo River and concluded that the instability in this reach is because the reconstructed channel is too narrow and steep. A full-scale survey of the former Plainwell impoundment would be needed to determine what changes have occurred since the TCRA was complete and the last repairs were made, and what types of repairs are needed for each location based on local conditions and flow characteristics (i.e. shear stress).

A significant number of roads and other TCRA-related features were left in-place following completion of the TCRA because it was anticipated that they would be needed in order to finish remedial obligations as part of floodplain soil alternative FPS-4a, which is the selected remedy for floodplain soils in the September 2015 Record Of Decision (ROD). The 7-acres of floodplain excavation outlined in the ROD has been significantly reduced based on results of the PDI and the Report concludes limited remedial actions in the former Plainwell impoundment floodplains may be necessary, including repair of failed banks and excavation of select areas. Although, as previously mentioned, EGLE believes additional work beyond what is described in the Report is likely necessary due to low data bias in the PDI data. The Michigan Department of Natural Resources (MDNR) has requested that roads and other features be removed as part of the remedial action in the former Plainwell impoundment. MDNR is contemplating doing additional work in the former Plainwell impoundment, the scope of which has not yet been finalized. EGLE appreciates GP and MDNR discussing and coordinating remedial actions and proposed projects in the former Plainwell impoundment. EGLE would encourage GP to continue to engage cooperatively with MDNR on response actions and proposed projects in the former Plainwell impoundment and requests that all final decisions related to the determination and removal of all TCRA-related features be coordinated with MDNR.

If you have any questions, please contact Mr. Daniel Peabody, Environmental Quality Analyst, Remediation and Redevelopment Division at 517-285-3924; PeabodyD@Michigan.gov; or EGLE, P.O, Box 30426, Lansing, Michigan 48909-7926

Sincerely,

Daniel Peabody
Environmental Quality Analyst
Remediation and Redevelopment Division

Enclosure
cc/enc: Mr. Mark Mills, Michigan Department of Natural Resources
Dr. Keegan Roberts, CDM Smith
Ms. Sydney Ruhala, EGLE
Mr. Joe Walczak, EGLE
Mr. Theo Von Wallmenich, Jacobs

Kalamazoo River Superfund Site
Pre-Design Investigation Evaluation Report Part 2
Near TCRA Area Floodplain Soil

GENERAL COMMENTS
Commenting Organization: EGLE **Commenter:**

General Comment #1: The report acknowledges that there are significant differences between the historical and the 2017/2018 data. As written, the language used suggests that the historical data is less representative of site conditions than the recent 2017/2018 data. However, the reasons provided (see specific comments below) are not well developed in the text and in some instances are misleading. It should be noted that Environmental Standards did not identify any historical data that should be rejected and states that for data where the historical guidance documents are available, it was determined that the data was analyzed and validated in accordance with the associated guidance document at that time (Appendix D). Environmental Standards acknowledges that the historical data should be used with caution, but they do not state that the data should rejected or ignored. The report should more clearly acknowledge that the reason(s) for the differences in historical and recent data is not fully known and should be considered when making remedial decisions moving forward. Further, as noted in Section 2.3, EGLE collected split samples from 22 parent samples from the 2018 data. Of the 22 splits, 10 were analyzed for Aroclors, while 12 were analyzed for Aroclors and congeners. EGLE's total polychlorinated biphenyl (PCB) concentrations via Aroclor analysis ($TPCB_{AROCLOR}$) were on average approximately 1.5 times higher than Wood's $TPCB_{AROCLOR}$ concentrations for the same sample. EGLE's average and median total PCB concentrations via congener analysis ($TPCB_{CONGENER}$) were approximately 2.4 and 2.0 times higher, respectively, than Wood's $TPCB_{AROCLOR}$ concentrations for the same sample. While this is a limited dataset, it indicates that the 2018 Aroclor data may be underrepresenting the amount of PCBs currently present in the near TCRA area floodplain soils (similar to the low bias observed in Pace Analytical Aroclor data in Area 4). In 2019, EGLE split 30 samples with Wood from the Area 4 floodplain. For the Area 4 samples, EGLE's $TPCB_{CONGENER}$ concentrations were on average approximately 2.3 times higher than Wood's $TPCB_{AROCLOR}$ concentrations for the same sample.

SPECIFIC COMMENTS
Commenting Organization: EGLE **Commenter:**

Section: 3.1 PCB Distribution **Page #:** 3-4
Specific Comment #1: In Section 3.1, the report acknowledges that there are significant differences between the historical data and the 2017/2018 data and provides several potential reasons for the differences. Three of the provided reasons are "improved field methods," "improved laboratory methods," and "improved survey methods". By using the term "improved", the text applies that the new 2017/2018 data is more representative of site conditions than the historical data, however this has not been demonstrated to date. Please change the word "improved" to "changes in" for each reason provided.

To-date GP has not provided any data or scientific explanation as to why "historic" data are inferior and continues to suggest that the "new" data is better simply because it is "new". That in and of itself is not a sufficient reason to ignore and not use "historic" data, which is the trend

in nearly all Areas of OU5. EGLE has performed statistical analyses on the data to look at data quality. EGLE's Area 1 Split Sample Evaluation, Test For Differences, and Congener and Aroclor Split Soil Samples Memoranda (attached) show that the "historic" data collected by multiple parties (including GP, EPA, and EGLE) and analyzed by multiple laboratories were consistent over time; only the "new" data collected by GP during the PDI is "unique", and; the 2018 Aroclor data may be underrepresenting the amount of PCBs currently present in the near TCRA area floodplain soils.

A separate analysis completed by EPA using Area 4 samples showed that GP's laboratory method is biased substantially low when compared to $TPCB_{AROCLOR}$ concentrations in EPA split samples and $TPCB_{CONGENER}$ concentrations in EGLE split samples. These analyses do not support the statements that the "new" data is "improved" and suggest the opposite.

Rather than arguing which data is "better", subjective statements should simply be removed from this section and throughout the document. EGLE continues to believe all data should be used for decision making purposes, documents will need to include realistic discussions about data quality, and a path forward to resolve data quality and comparability issues will be necessary.

Section: 3.1 PCB Distribution **Page #:** 3-4
Specific Comment #2: In Section 3.1, the report also provides "time elapsed since historical samples were collected" and a potential cause for the significant differences between the historical data and the 2017/2018 data. However, in Section 3.2, the report states that Environmental Standards saw no signs of PCB weathering during their evaluation of the 2017/2018 data. If the PCBs have not weathered over time and are known to be environmentally persistent, please provide examples of how elapsed time between sample collections may have resulted in the significant differences between the historical data.

The State appreciates GP hiring Environmental Standards Inc. (ESI) to perform a review of data quality on their behalf and conclude that the data GP collected during the PDI are of "high quality" and "show no signs of weathering or directional bias". EGLE notes that these statements are inconsistent with previous site documents authored by multiple parties (including GP) and statements made by GP during the 2015 bench trial. EGLE has also authored several comment letters and technical memoranda clearly documenting low bias in GP's $TPCB_{AROCLOR}$ data, detailing our conceptual model of what is occurring, and outlining at least one potential path forward to address $TPCB_{ARCOLOR}$ bias. EGLE appreciates the EPA opening up a dialogue amongst the Work Group and providing the State the opportunity to participate in discussions with all Parties about data quality and determine what (if any) corrective measures need to be taken moving forward.

Section: 3.2 Data Quality Evaluation **Page #:** 3-5
Specific Comment #3: In Section 3.2, the report states that Environmental Standards found that the historical data was "collected for different purposes, by different entities, and were analyzed by different laboratories, some of which are no longer in operation, and one that was closed for improper analytical techniques." EGLE has previously provided documentation demonstrating that the Southwest Laboratory of Oklahoma was closed for improper analytical techniques specifically regarding Method 8260 for Volatile Organic Carbons (VOCs). Please add "for

Method 8260 for VOCs" to the end of the sentence or remove the language "and one that was closed for improper analytical techniques" since the text is currently misleading as written.

Section: 3.4 Moving Window Analysis **Page #:** 3-6
Specific Comment #4: In Section 3.4, the report states that "Four home range scenarios were evaluated for the remedial design: 1 acre for the 0- to 6-inch interval (representing the house wren) (Figures 3-8a and 3-8b)…". However, Table 3-2 states that a 2-acre home range was used for the 0- to 6-inch interval. Figures 3-8a and 3-8b also show a 2-acre home range for the 0- to 6-inch interval. Please revise to be consistent.

Section: 3.6 Post-TCRA Bank Monitoring Findings **Page #:** 3-6
Specific Comment #4: In Section 3.6, the report states that "During the 2015 monitoring event, MDNR/EGLE observed water flowing behind restored banks in two areas on the left-descending bank (LDB), downstream of the US-131 Bridge, indicating evidence of bank erosion in these locations. MDNR/EGLE observed water flowing behind restored banks in a third area in 2016 and a fourth area in 2018, also on the LDB downstream of the US-131 Bridge. Wood confirmed the locations of these findings in September 2019". EGLE notes that erosion within the monitored area had been documented and "repairs" to the banks had to be made prior to the Michigan Department of Natural Resources (MDNR)/EGLE taking over post-TCRA monitoring.

Section: Figure 3-13 and Appendix F MDNR/EGLE Bank Monitoring Reports **Page #:**
Specific Comment #5: EGLE believes there are other erosional areas within the former Plainwell impoundment that are not documented in EGLE's bank monitoring reports. For example, EGLE's bank monitoring effort did not include any monitoring of the right descending bank (RDB) and erosion has been observed above and below the US-131 bridge along the RDB. A complete survey of the former Plainwell impoundment followed by a thorough analysis using as-built drawings, yearly monitoring reports and other data sources, would be necessary to determine the full extent of erosion that has occurred and what potential remedies to achieve bank stabilization at each location may be necessary. EGLE also performed a 2019 Bank Monitoring Survey that is not included in the Report.

Following the TCRA, land within the TCRA boundary (some of which was covered with topsoil and revegetated) was not maintained and many of the plantings died. As a result, invasive species moved in and now dominate the floodplain landscape. During remedial actions in these locations, care should be taken to prevent the further spread of invasive species, areas backfilled should be properly restored with native and desirable vegetation in coordination with the landowner (MDNR), and a post-construction vegetation cover survey of the floodplain should be done to determine if taking active measures to control invasive species is necessary.

Memorandum

To: *Danial Peabody*

From: *Scott Kirchner, CHMM*

Date: *15 October 2018*

Subject: *Working Draft Summary of Area 1 Split Sample Evaluation*

CDM Smith was tasked to accept and analyze split samples for Aroclor analysis by EPA Method 8082A. The samples were collected during the pre-design sampling event conducted by Wood Environment & Infrastructure Solutions, Inc. (Wood) on behalf of Georgia Pacific (GP) at the Area1 OU-5 Allied Paper, Inc., Portage Creek Kalamazoo River Superfund Site (the site), performed between 19 June through 27 June 2018.

CDM Smith accepted split aliquots of 23 environmental soil samples, one duplicate and one sample for matrix spike and matrix spike duplicate analyses. All samples were shipped under a single chain of custody to Integrated Analytical Labs (IAL) located at 273 Franklin Road, Randolph, NJ 07869. CDM Smith also submitted three certified reference material (CRM) samples under separate chain of custody to be analyzed along with the environment samples. A summary of the samples submitted to LAI is provided in the attached data validation report.

Summary of Split Sample Finding

As a general rule of thumb a relative percent difference (RPD) of less than 50 between soil sample split results is considered acceptable. The RPDs of the individual Aroclors and total Aroclor results reported by each laboratory are all less than 50, Table 1. While these results are encouraging the results of the split samples (MDEQ) tend to trend higher than those of GP. Under a normal distribution scenario the results should be more evenly dispersed around a one to one line as noted in *Section 1.3* and *Figure 8* of the attached *Test for Differences in Split and Paired Samples* report. As indicated in this report the total Aroclor concentrations from the 2017 and 2018 pre-design investigation (PDI) samples collected by Wood in the formerly impounded floodplains of OU5/Area 1 were unexpectedly lower than total Aroclor concentrations in samples collected from this same area in 2008, 2001 and 1993/1994 by USEPA, MDEQ and Georgia-Pacific.

Additionally, the majority of the split sample surrogate recoveries and the CRM recoveries tended to be on the low end of the acceptable range. This lead us to believe that the results from the Aroclor analysis are generally biased low. Due to this MDEQ has elected to submit several samples for analysis by EPA method 1668 *Chlorinated Biphenyl Congener in Water, Soil, Sediment, Biosolids,*

and Tissue by HRGC/HRMS, EPA-820-R-10-005. These samples are currently under analysis and the findings will be amended to this report.

Analytical Methodology

EPA Method 8082A is a gas chromatography method that uses comparison to a standard cure and Aroclor pattern recognition to quantify and identify Aroclors from a 30 gram soil subsample. Surrogates are added to each sample prior to extraction as a means to evaluate extraction and analytical efficiency. IAL used methylene chloride as an extraction solvent and extracted the samples via ultrasonic extraction technique.

cc: Dr. Keegan Roberts

1 Total Aroclors in Area 1 Formerly Impounded Sediments

Total Aroclor concentrations from the 2017 and 2018 pre-design investigation (PDI) samples collected by Wood Environment & Infrastructure Solutions, Inc. on behalf of Georgia-Pacific in the formerly impounded floodplains of OU5/Area 1were unexpectedly lower than total Aroclor concentrations in samples collected from this same area in 2008, 2001 and 1993/1994 by USEPA, MDEQ and Georgia-Pacific. Pre-2017 data for this Area have consistently shown that low lying areas (e.g., old river channels and low flat terraces) have total Aroclor concentrations averaging approximately 10 mg/kg or more. These same data also commonly exceeded total Aroclor concentrations of 20 mg/kg, with occasional exceedances of 50 mg/kg (i.e., TSCA material). The Aroclor concentrations in the pre-2017 data are spatially heterogeneous, but with some predictable trends including thicker sediment deposits with higher concentrations within old thalwegs. Temporal trends over the last 30 years in these Aroclor concentrations are not expected because concentrations are generally lower than those for which substantive bio-degradation might be occurring, the PCBs are bound with highly organic recycled paper residuals, and PCBs have been shown to be environmentally persistent organic pollutants (POPS).

Contrary to this understanding of fate and transport of Aroclors in OU5/Area 1, the total Aroclor concentrations in PDI samples (collected in 2017 and 2018)are distinctly lower than those in the RI/FS data (collected from 1993 through 2008) (Figure 1 Figure 2). In an effort to understand this data inconsistency, a series of investigations was conducted, and are identified below:

1) Understanding of temporal changes in total Aroclors in floodplain soils was based on statistical analyses conducted by Kern 2001. These analyses indicated no differences in total Aroclor distributions between 1993/1994 and 2001. These analyses were reviewed and key graphics reproduced in this report.
2) It was suggested that preferential sectioning of cores based on stratigraphy (e.g., focused collection of sediment layers exhibiting visual clues for contamination) could explain apparent bias between PDI and RI/FS data. Consequently, a comparison of total Aroclors was conducted for cores that were a.) in close proximity (i.e., 15 feet) of each other and b.) were sectioned consistently. This comparison was to determine if there were differences in total Aroclors that could not be explained by these differences in segmenting the cores or core handling procedures.
3) Differences in laboratory analytical methods were evaluated by comparing split samples sent to the PDI lab used by Georgia Pacific and an alternative laboratory used by MDEQ.
    a. Standard certified reference material (CRM) prepared by Environmental Resource Associates (ERA) was also sent to the MDEQ laboratory to evaluate accuracy of reported Aroclors in the MDEQ split samples.

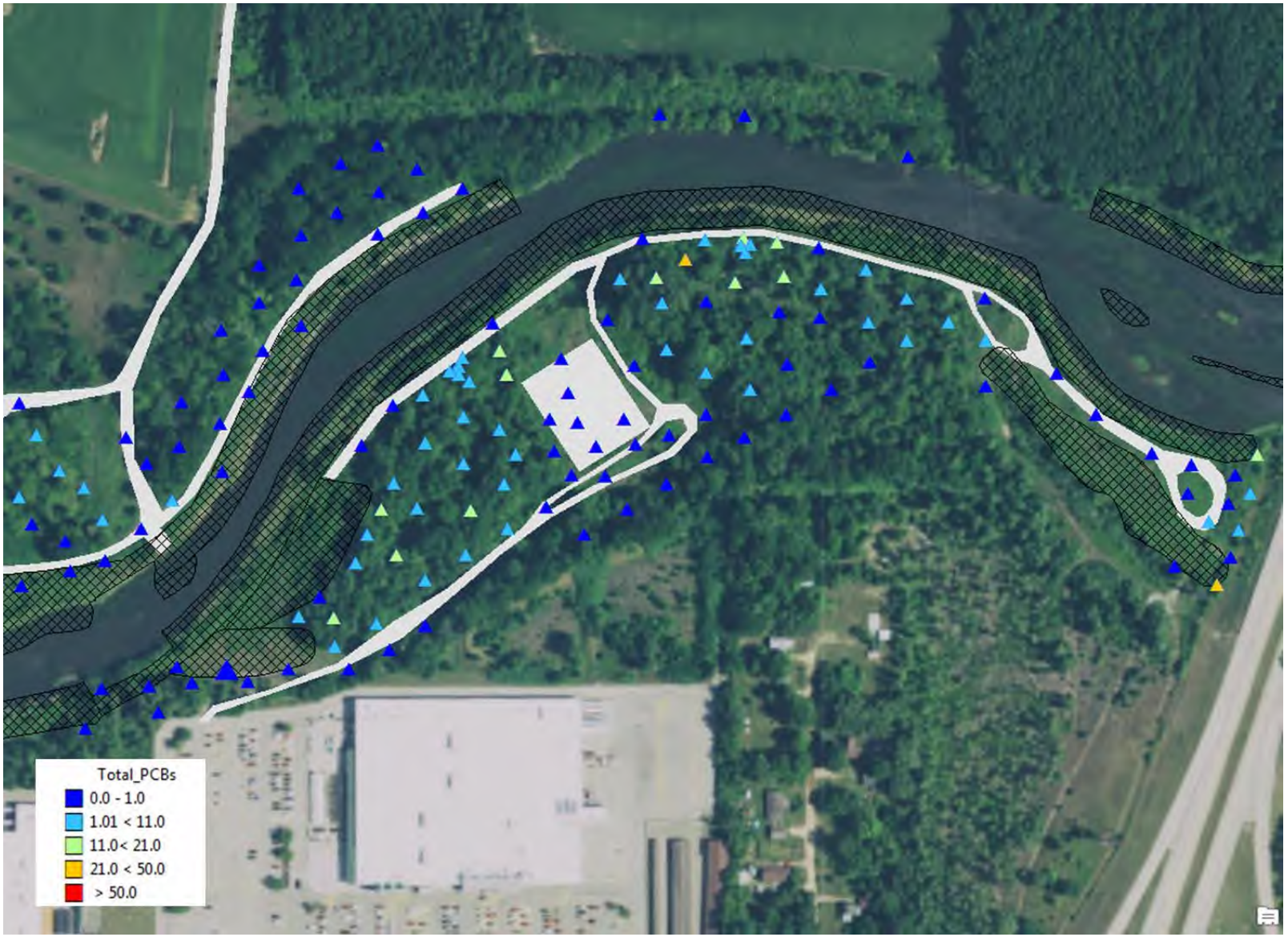

Figure 1. Total Aroclor concentrations in 0 to 6 inch depth interval for samples collected in 2017 and 2018 pre-design investigation.

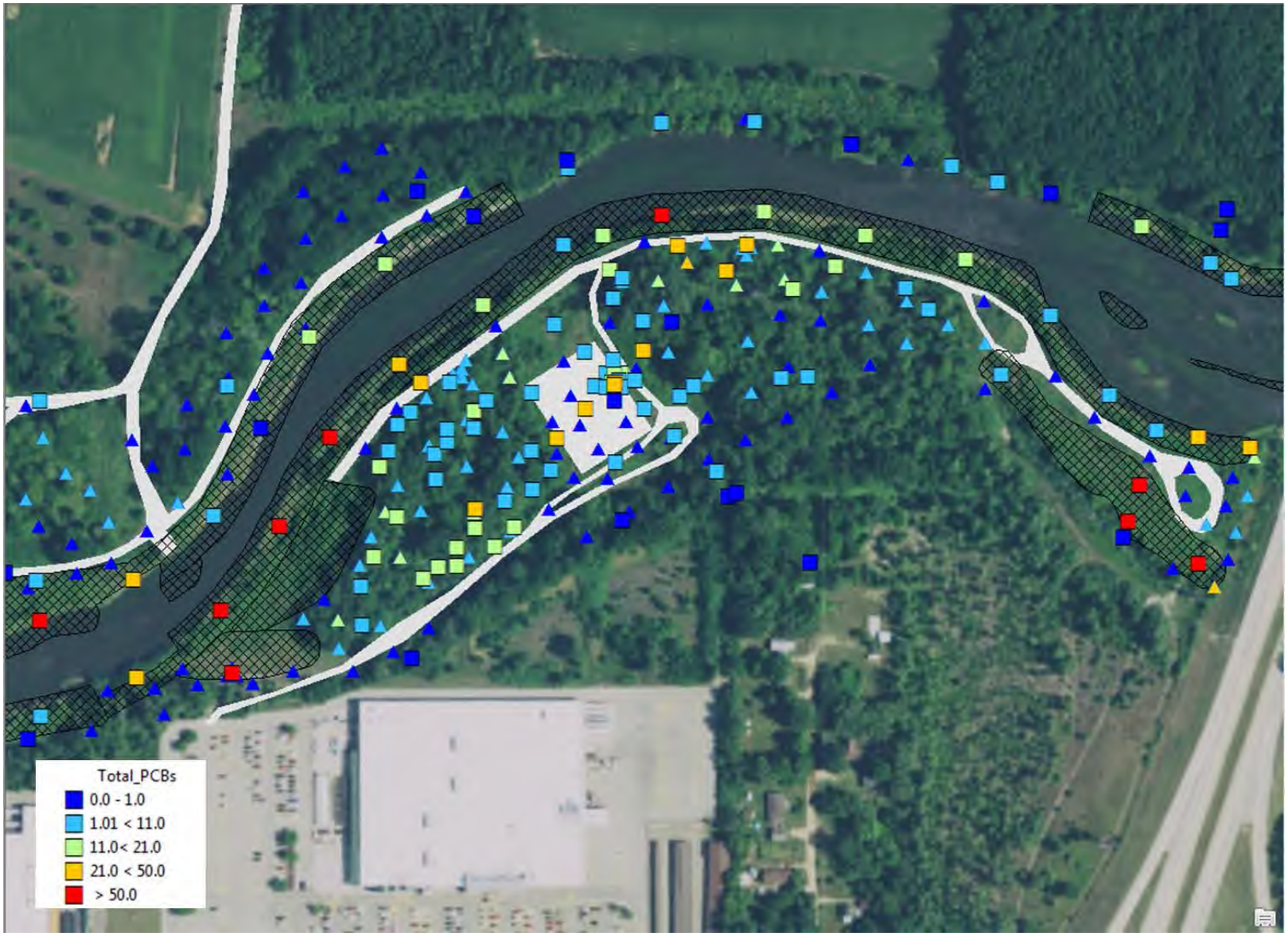

Figure 2. Total Aroclor concentrations in 0 to 6 inch depth interval for samples collected in 2017 and 2018 pre-design investigation (triangles) and in RI/FS investigations (squares)

1.1 Temporal Trends from 1993 to 2001

Kern (2001) conducted an analysis comparing PCB Aroclor samples collected in 1993 with samples collected in 2001. The 1993 data were collected by the Blasland Bouck and Lee (BBL) on behalf of Georgia Pacific, and the 2001 data were collected by Weston Solutions on behalf of the United States Environmental Protection Agency, with limited split samples collected by the Michigan Department of Environmental Quality also in 2001. These data were subjected to careful data handling and statistical analyses by Kern Statistical Services to evaluate potential temporal trends in total Aroclors in exposed formerly impounded sediments and instream sediments at Plainwell and Otsego City Impoundments. Kern (2001) found that the median and statistical distributions of formerly impounded and instream sediments were not statistically different, and that total Aroclors from split samples collected by the MDEQ and USEPA were variable, but exhibited little or no bias. As can be seen from the following figure split samples are evenly spread around a regression line with slope 0.97 which is nearly identical to the 1:1 line, representing unbiased measures.

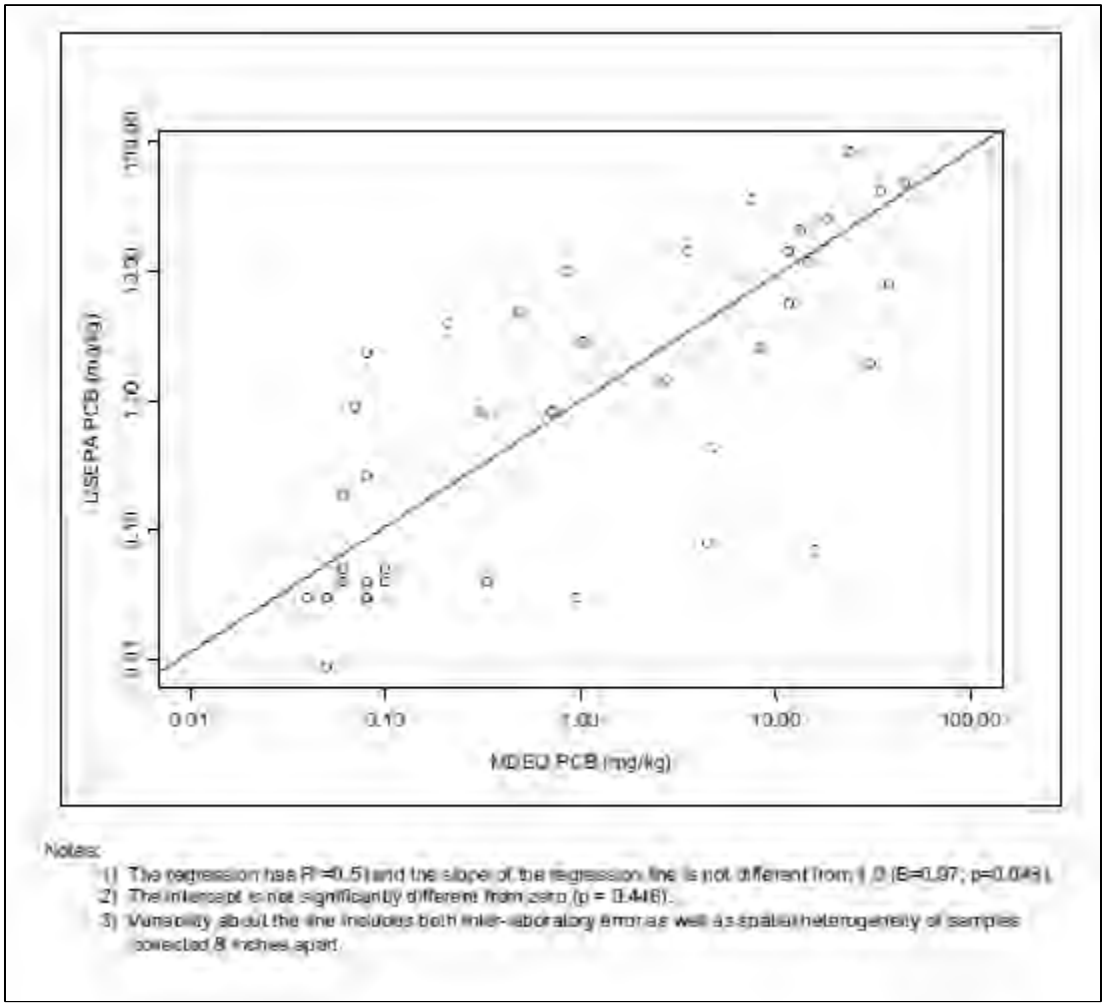

Figure 3. USEPA total Aroclors vs MDEQ total Aroclors collected in 2001 from Plainwell Impoundment and Otsego City Impoundments (Reproduced from Kern, 2001).

The distribution of formerly impounded sediments from these three sets of samples are displayed as side by side boxplots in **Figure *4***. This figure shows that the 25th and 75th percentiles (top and bottom of gray boxes) are nearly identical and that confidence intervals (notches in boxes) median total Aroclor concentrations (red horizontal line) are strongly overlapping. Statistical tests performed in 2001 indicate these median concentrations do not differ statistically (Kruskall Wallace; p= 0.41). In the Kern report this comparison was reported as evidence of comparability because there was general agreement that there would be no trends in total PCBs in floodplain soils because PCBs are persistent pollutants and do not generally degrade perceptibly even over very long periods of time.

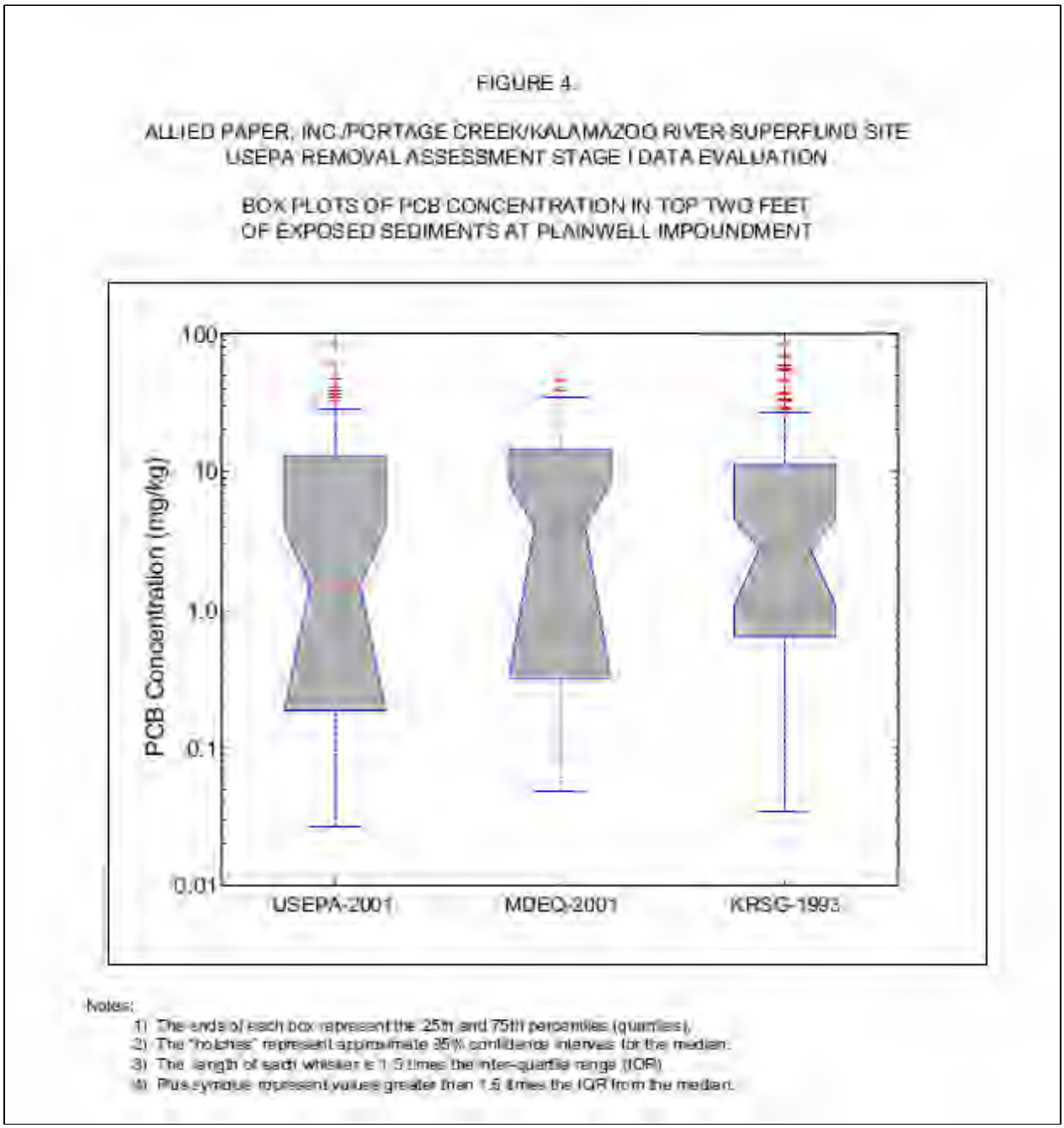

Figure 4. Comparison of 1993 and 2001 total Aroclors reported by Kern Statistical Services (2001).

1.2 Aroclors in Similarly Sectioned Proximal Core Samples

Core samples from the RI/FS were paired spatially with PDI cores within 15 feet and 20 feet. These pairs were examined to identify instances where core sections in both locations were sectioned regularly into 0 to 6, 6 to 12, 12 to 24 inch intervals. This pairing of core sections for which both cores were sectioned

identically provided a control for any bias related to preferential sectioning of cores in the RI/FS data. These paired core sections were plotted in three ways:

1) Total Aroclors in the PDI samples were plotted against total Aroclors in the RI/FS samples and 1 to 1, 2 to 1, 10 to 1 and 100 to 1 lines were overlaid on the plots (Figure 5). Equivalent analyses would be indicated by an equal scatter of sample pairs above or below the 1 to 1 line.
2) Ratios of the RI/FS samples divided by PDI samples were plotted:
    a. Against distance between pairs
    b. Against concentration in the RI/FS samples

1.2.1 PDI vs RI/FS Paired Core Sections

Paired PDI and RI/FS total Aroclors are plotted in Figure 5 showing that for pairs of cores sectioned identically and within 15 feet of each other resulted in substantively lower Aroclor concentrations in PDI core sections as compared with RI/FS cores. For these paired core sections, all but one resulted in the RI/FS sample having higher concentrations than the PDI samples. The RI samples were generally on the order of a factor of 2 higher than the PDI samples with some instances where the RI/FS total Aroclors were a factor of 10 higher or more. This systematic bias between the sampling and analysis programs confirms the apparent differences in maps of RI/FS vs PDI sample results. Further it is notable that factor of 2 differences are observed at both the low and high end of the concentration range. Particularly important are factor of 2 or greater ratios for samples of 10 mg/kg and 20 mg/kg which can be expected to influence estimates of exposure in areas with concentrations close to the remedial action limits (RAL).

1.2.2 Ratio of PDI to RI/FS total Aroclors

The ratio of total Aroclor concentration in RI/FS samples to PDI samples are plotted against distance between core location in (Figure 6). The ratios are greatest for sample pairs within approximately 15 feet with values ranging from just over 1 to 1 to as much as 50 to 1. The ratios are more or less randomly distributed for distances greater than about 15 feet which is expected regardless of the degree of agreement between sample values because spatial heterogeneity in the concentrations is expected to cause paired values to be independent and therefore the ratios should be randomly distributed about the value of 1.0.

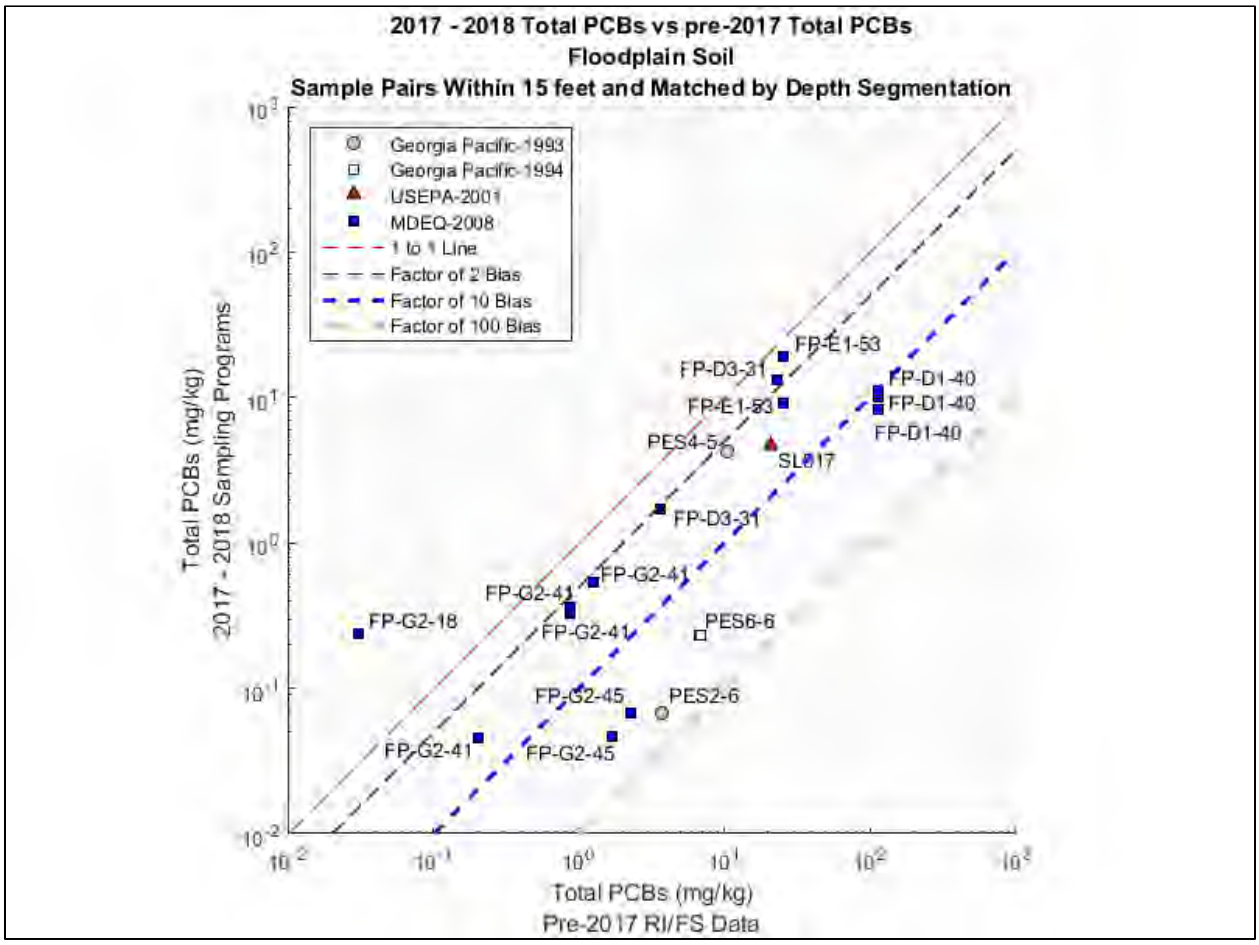

Figure 5. Total Aroclors in PDI samples vs total Aroclors in RI/FS samples for cores sectioned identically and within 15 feet in proximity.

The ratios of PDI to RI-FS samples are also plotted against total Aroclors in the RI/FS samples showing that the ratio generally increases with concentration in the RI samples, and that for RI samples with total Aroclors exceeding 15 mg/kg all ratios are greater than 1 with most ratios greater than 2 and with several ratios exceeding 10 (Figure 7). This indicates that at locations sampled in 2017 and 2018 and forming the basis for mapping total Aroclors and determining exposure, mapped values may differ from maps developed for the RI/FS by a factor of 2 or more.

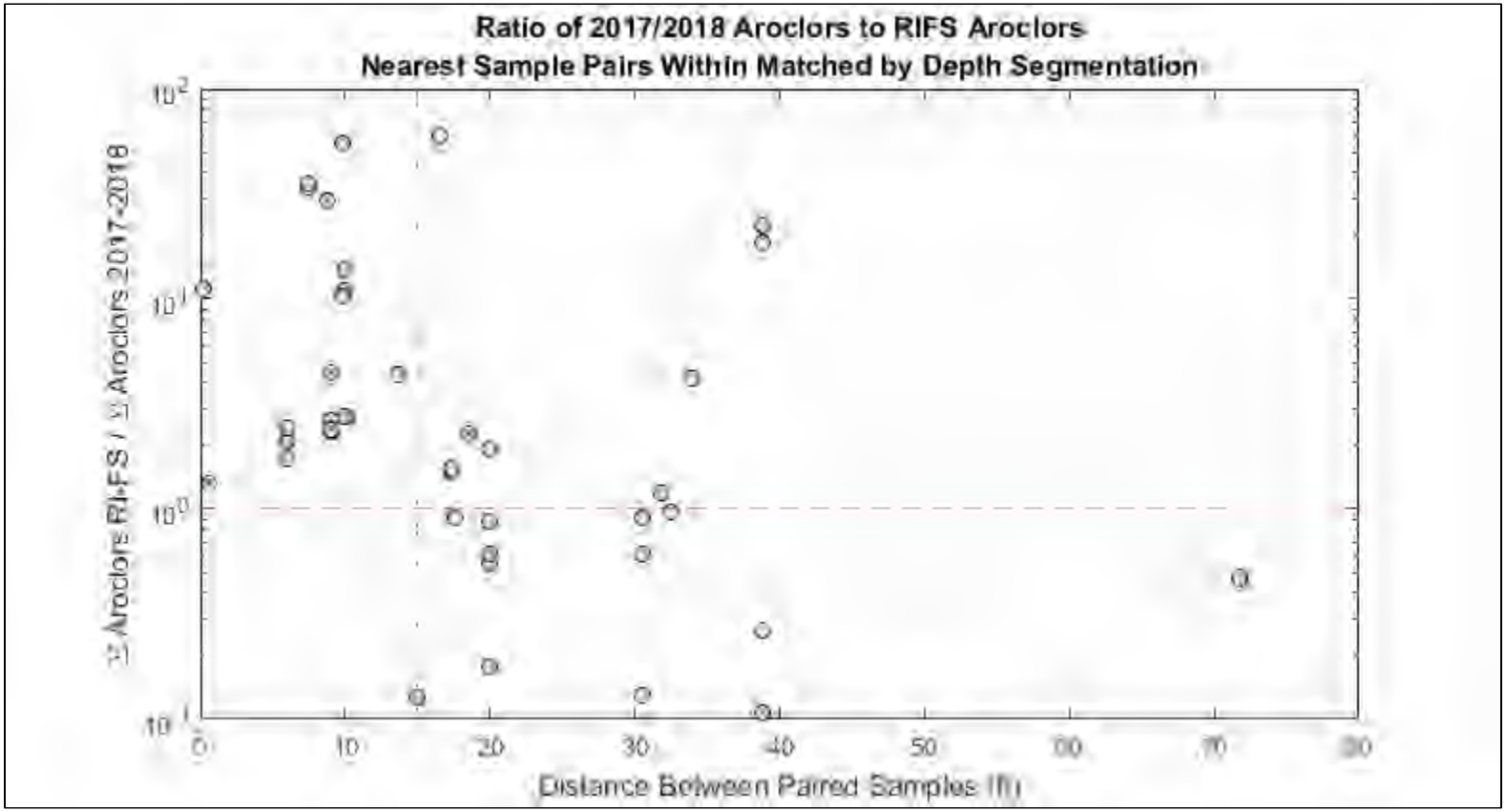

Figure 6. Ratio of PDI to RI/FS total Aroclors against distance between core locations.

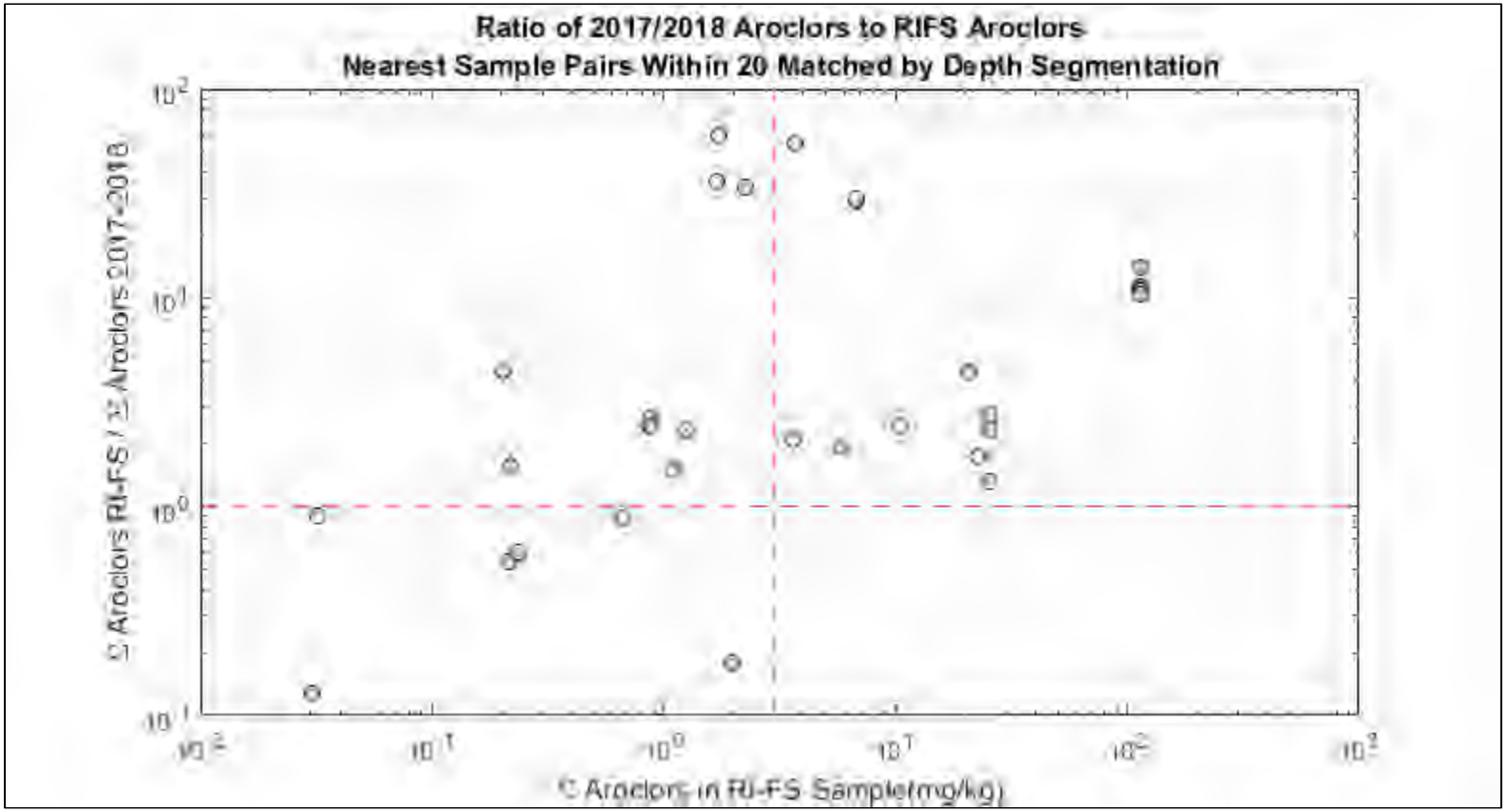

Figure 7. Ratio of PDI to RI/FS total Aroclors against total Aroclor concentratoin in RI-FS sample.

1.3 Laboratory Split Samples

Twenty-two samples were spit and analyzed by both MDEQ and Wood to test for inter-laboratory differences in total Aroclor quantitation. The MDEQ laboratory reported more Aroclors (Aroclors 1262 and 1268) contributing to the totals than the Wood laboratory, so total Aroclors were compared in two ways; first based on total of all Aroclors reported by each laboratory and secondly based only on the total of Aroclors reported by the Wood laboratory. Some statistical results were sensitive to this data handling choice.

When considering the full set of Aroclors, values reported by MDEQ exceeded those reported by Wood in 15 of 20 pairs, whereas when restricting the MDEQ totals to only those Aroclors reported by Wood, the MDEQ totals exceeded the Wood totals in 13 of 20 cases. On average, for all Aroclors, the ratio of MDEQ to Wood total Aroclors was R=1.5 (CI: 1.4, 1.6). For the restricted set of Aroclors reported by Wood, the ratio was R=1.4 (CI: 1.1, 1.7). Because the lower confidence limits are greater than 1.0, one can conclude that total Aroclor concentrations reported by Wood are less than those reported by MDEQ by approximately a factor of 1.5 (1.4 for restricted set of Aroclors) with 95% level of confidence. These analyses are presented in Figure 8 and on Table 1.

The pairs were also subjected to nonparametric sign test which tests the null hypothesis that the median concentrations of each set of data are equal, based on a paired statistical design. For the full set of Aroclors, the sign test indicated differences in medians at the 95% level of confidence, whereas for the reduced set of Aroclors, median total Aroclors were not found to differ.

Summarizing, these results are not fully consistent, although for three of four evaluations (Ratios, full and reduced Aroclor set, and sign test full set of Aroclors) the tests indicated statistical differences and on average the total Aroclors reported by Wood were on the order of 50% lower than those reported by MDEQ for the full and reduced set of Aroclors.

Michigan DEQ also submitted a CRM performance standard to the analytical lab and found that the reported value was on the low end of ERA's QC performance acceptance limits (QC-PAL), which suggests that the low bias identified in split samples is indicative of inaccuracy in the laboratory results reported by Wood. Table 2 contains a summary of MDEQ's CRM sample recoveries as compared to ERA's QC-PAL, mean reported result and true value of the associated Aroclor.

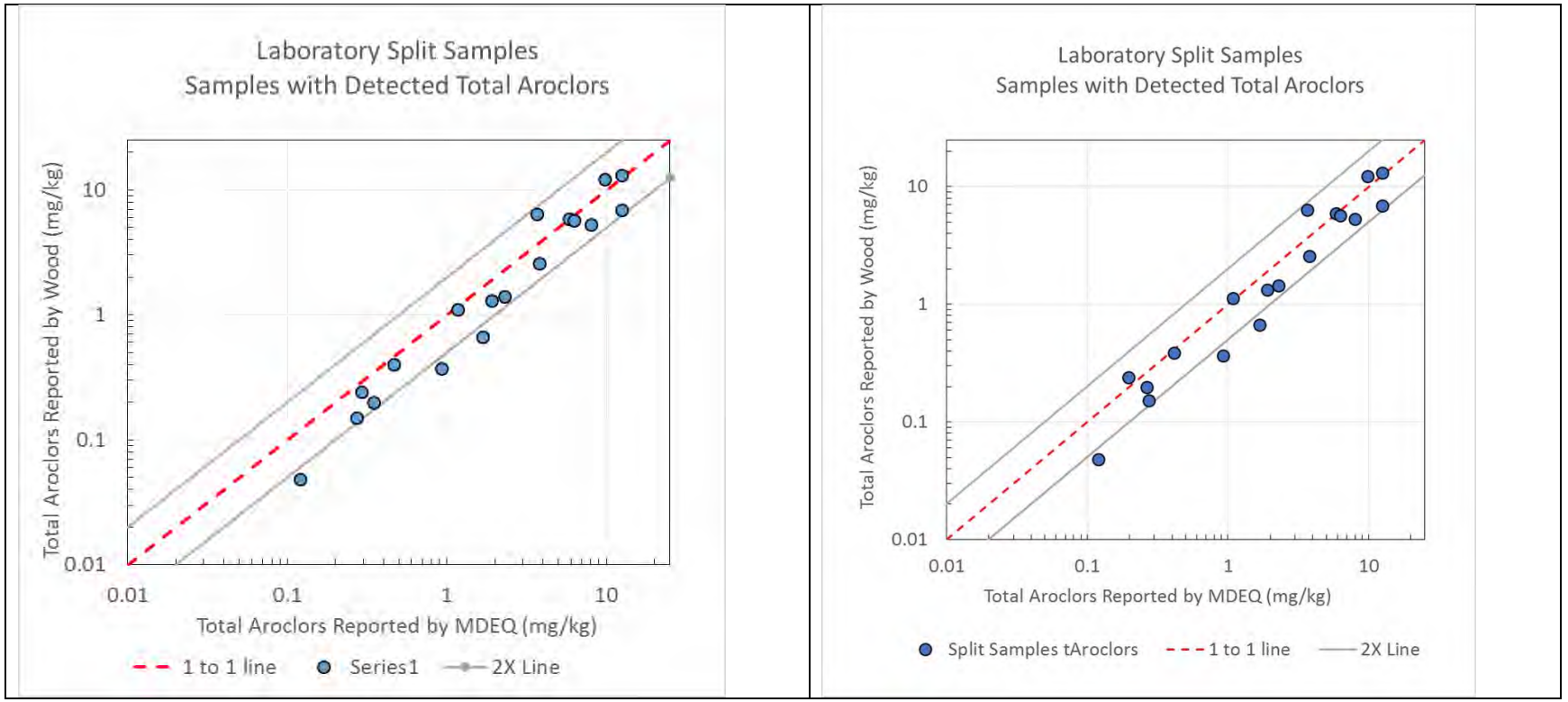

Figure 8. Total Aroclors reported by Wood vs reported by MDEQ in split samples for the full set of Aroclors (left panel) and for the restricted set of Aroclors (right panel).

Table 1. Split sample total Aroclor results for Plainwell Impoundment floodplain soils.								
			Total Aroclors MDEQ (mg/kg)		Total Aroclors Wood (mg/kg)			
sys_loc_code	Start Depth	End Depth	Full Aroclor Set	Reduced Aroclor Set	Reduced Aroclor Set	Ratio (Full) (MDEQ/Wood)	Ratio (Reduced) (MDEQ/Wood)	DEQ > Wood?
A1-FPS-142	0	6	5.86	5.86	5.89	0.99	0.99	0
A1-FPS-144	6	12	0.292	0.197	0.24	1.22	0.82	0 (1)
A1-FPS-146	12	24	6.29	6.29	5.72	1.10	1.10	1
A1-FPS-153	6	12	0.349	0.264	0.198	1.76	1.33	1
A1-FPS-178	12	24	0.014	<0.011	<0.042		No Estimate	
A1-FPS-183	0	6	0.713	0.606	<0.042		No Estimate	1
A1-FPS-183	12	24	1.9	1.895	1.32	1.44	1.44	1
A1-FPS-188	6	12	<0.011	<0.011	<0.042		No Estimate	
A1-FPS-196	6	12	<0.009	<0.009	0.06		No Estimate	0
A1-FPS-201	6	12	0.12	0.12	0.048	2.50	2.50	1
A1-FPS-206	6	12	1.17	1.09	1.12	1.04	0.97	0(1)
A1-FPS-213	6	12	3.65	3.65	6.35	0.57	0.57	0
A1-FPS-217	0	6	12.5	12.46	13.2	0.95	0.94	0
A1-FPS-236	12	24	0.273	0.273	0.152	1.80	1.80	1
A1-FPS-246	6	12	0.465	0.414	0.389	1.20	1.06	1
A1-FPS-256	0	6	9.81	9.809	12.2	0.80	0.80	0
A1-FPS-281	12	24	1.68	1.677	0.67	2.51	2.50	1
A1-FPS-304	6	12	0.926	0.926	0.364	2.54	2.54	1
A1-FPS-312	0	6	3.78	3.781	2.56	1.48	1.48	1
A1-FPS-316	0	6	8.02	8.02	5.33	1.50	1.50	1
A1-FPS-338	0	6	2.29	2.292	1.44	1.59	1.59	1
A1-FPS-351	6	12	12.5	12.53	6.9	1.81	1.82	1

Table 2 MDEQ Summary of Recoveries of Certified Reference Material

Sample #:		CRM PCB IN SOIL 1248-496				CRM PCB IN SOIL 1260-494				CRM PCB IN SOIL 1254-492			
Field ID:													
Lab ID:		05171-025				05171-026				05171-027			
Date Sampled:		06/27/2018				06/27/2018				06/27/2018			
Depth(ft):		True Value =		4.91	Mean	True Value =		3.19	Mean	True Value =		3.31	Mean
	CAS	QC Range (n=66)		2.24 - 6.19	2.98	QC Range (n=62)		1.38 - 3.73	2.59	QC Range (n=67)		1.36 - 4.04	2.65
PCB's (mg/Kg)		Conc	Q	RL	MDL	Conc	Q	RL	MDL	Conc	Q	RL	MDL
Aroclor-1016	12674-11-2	ND		0.00168	0.000672	ND		0.00168	0.000672	ND		0.00167	0.000668
Aroclor-1221	11104-28-2	ND		0.00168	0.000672	ND		0.00168	0.000672	ND		0.00167	0.000668
Aroclor-1232	11141-16-5	ND		0.00168	0.000672	ND		0.00168	0.000672	ND		0.00167	0.000668
Aroclor-1242	53469-21-9	ND		0.00168	0.000672	ND		0.00168	0.000672	ND		0.00167	0.000668
Aroclor-1248	12672-29-6	2.29	D	0.017	0.00673	ND		0.00168	0.000672	ND		0.00167	0.000668
Aroclor-1254	11097-69-1	ND		0.00168	0.000672	ND		0.00168	0.000672	2.22	D	0.033	0.013
Aroclor-1260	11096-82-5	ND		0.00168	0.000672	1.86	D	0.017	0.0067	ND		0.00167	0.000668
Aroclor-1262	37324-23-5	ND		0.00168	0.000672	ND		0.00168	0.000672	ND		0.00167	0.000668
Aroclor-1268	11100-14-4	ND		0.00168	0.000672	ND		0.00168	0.000672	ND		0.00167	0.000668
Total	1336-36-3	**2.29**	D	0.017	0.00673	**1.86**	D	0.017	0.0067	**2.22**	D	0.033	0.013
		76.85% %		Recovery from mean		71.81% %		Recovery from mean		83.77% %		Recovery from mean	
		46.64% %		Recovery from true value		58.31% %		Recovery from true value		67.07% %		Recovery from true value	
				Recovery of mean	60.60%			Recovery of mean	81.30%			Recovery of mean	80.10%

ND – nondected Q – lab qualifier RL – reporting limit MDL – method detection limit

D – result from diluted sample analysis

Memorandum

To: *Daniel Peabody*

From: *Scott Kirchner, CHMM*

Date: *November 28, 2018*

Subject: *Summary of Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site, Operable Unit 5, Area 1 Polychlorinated Biphenyl Congener and Aroclor Split Soil Samples*

Introduction

This memorandum summarizes polychlorinated biphenyl (PCB) congener and Aroclor results from the Michigan Department of Environmental Quality's (MDEQ's) split soil sampling for Operable Unit 5 (OU-5) Area 1 of the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site (site). These samples were collected during the OU-5 Area 1 pre-design sampling event performed June 19 through 27, 2018.

During the 2018 soil sampling event, Wood Environment & Infrastructure Solutions, Inc. (Wood) personnel collected multiple soil samples. Wood personnel homogenized and split with MDEQ twenty-two of these soil samples and one field duplicate. MDEQ accepted two aliquots of each split sample, one for Aroclor analysis and one that was held by its analytical laboratory for potential PCB congener analysis. Wood's split soil samples were sent to Pace Analytical Services, LLC (Pace) of Green Bay, Washington for Aroclor extraction and analysis. MDEQ sent all of its aliquots to Integrated Analytical Laboratories, LLC (IAL) of Randolph, New Jersey, with the first set of twenty-two soil splits subjected to Aroclor extraction and analysis. Following IAL's Aroclor analyses, CDM Smith personnel selected twelve of the split sample aliquots to be sent by IAL to Vista Analytical Laboratory (Vista) of El Dorado Hills, California. Vista performed PCB congener extraction and analysis on these twelve split samples.

CDM Smith also submitted three certified reference material (CRM) samples to IAL to examine the laboratory's ability to identify and recover the known Aroclor concentration in the CRMs. CDM Smith also submitted a single CRM sample to Vista to be included in extraction and congener analysis. The extraction method used by Pace was EPA Method SW-846 3541 *Automated Soxhlet Extraction* (EPA,1994). The extraction method used by IAL was EPA Method SW-846 3550c *Ultrasonic Extraction* (EPA, 2007). Both Pace and IAL labs utilized EPA Method 8082A *Polychlorinated Biphenyls (PCBs) by Gas Chromatography* (EPA, 2007a). Vista used EPA Method 1668, Revision A *Chlorinate Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS* for extraction and analysis of 209 PCB congeners (EPA, 1999).

CDM Smith provided a memorandum discussing the 2018 Aroclor split sample results and IAL's associated data report to MDEQ on October 15, 2018 (CDM Smith, 2018). Wood, Georgia-Pacific, and International Paper subsequently requested Environmental Standards, Inc (ES) from Valley Forge, PA review the data package provided by MDEQ's contract laboratory, IAL. ES's November 5, 2018 memorandum stated that although there were differences in the Pace and IAL extraction methods, the split data compared well as all split sample relative percent differences (RPDs) met the QAPP criteria of 100% and 15 samples met MDEQ's field duplicate criterion of 50% (Environmental Standards, 2018).

Following receipt of IAL's Aroclor split sample results, MDEQ and CDM Smith directed IAL to send twelve of the split samples to Vista for congener analysis. The congener analyses were to be used to confirm that the reported total PCB Aroclor concentration results sufficiently represented total PCB concentrations present in the samples and, ultimately, at the site. The split sample results from both sets of Aroclor analyses (Pace and IAL) and PCB Congener analysis are presented below to advance the understanding of soil PCB concentration results in OU-5 Area 1 soils. A thirteenth split sample submitted for congener analysis may have been treated as a matrix spike and therefore is not being included as part of this evaluation.

Literature Summary of Aroclor versus Congener PCB Analysis

Ecological risk assessment has historically utilized Aroclor analysis for assessment of PCB contamination in environmental media. Aroclors are multi-component mixtures, so analysis of individual Aroclors in environmental media requires matching a sample's individual Aroclor patterns to those of the Aroclor standards used during the analysis (Bernhard & Petron, 2001; Butcher, Gauthier, & Garvey, 1997; EPA, 2007). One of the main challenges encountered during Aroclor analyses is the potential inability to identify individual Aroclor patterns. This could be due to:

- presence of multiple Aroclors,
- significant temporal changes in Aroclor patterns arising from:
    - weathered PCBs,
    - degraded PCBs,
    - metabolized PCBs

(Bernhard & Petron, 2001; Butcher et al., 1997; Cleverly, 2003; EPA, 2007; Rushneck et al., 2004; Stalling, Schwartz, Dunn, & Wold, 1987).

The inability to accurately identify individual Aroclor patterns can lead to inadequate data on total PCB concentrations, exposure, and risks, and ultimately to less or non-effective risk-based soil remediation efforts. An alternative to Aroclor analysis is congener analysis, where 209 individual PCB compounds (also known as congeners) are analyzed. Congener analysis provides the ability to identify total PCB concentration regardless of the source congener mixture or its complexity. Although congener analysis is more costly than Aroclor analysis, congener analysis has lower quantification limits, is not subject to trying to identify individual Aroclor patterns, and can provide more direct measurements of risk due to PCB toxicity being congener based (Bernhard & Petron, 2001; Cleverly, 2003; Narquis, Prignano, & Hyatt, 2007).

Split Sample Finding

MDEQ's split sample study was not designed to replicate Wood's analytical approach. The main objectives of MDEQ's soil split sampling for OU-5 Area 1 study were:

1. to confirm Wood's identification of site Aroclors using EPA Method 8082A,
2. to confirm Wood's reported concentration of total Aroclor PCB using EPA Method 8082A,
3. to evaluate if using EPA Method 8082A for total Aroclor results underrepresented total PCB concentrations at the site by comparison to total PCB by congener results using EPA Method 1668A.

Three sets of data are used in this evaluation:

1. Aroclor analysis under method 8082A performed by Pace following their analytical SOPs,
2. Aroclor analysis under method 8082A performed by IAL following their analytical SOPs,
3. congener analysis under method 1668A performed by Vista following their analytical SOPs.

Total PCBs

The soil results presented below are compared based on the total PCB concentrations reported by the analytical laboratories. This number is a sum of all individual concentrations detected, either as Aroclors or congeners detected. The IAL data included two additional Aroclors compared to Pace (Aroclors 1262 and 1268); however, the results for these two additional Aroclors were excluded when calculating total PCB concentration present to better directly compare the lab results as performed under method 8082A. As seen on **Figure 1** below, total PCBs for Aroclor analysis from both labs seem to be comparable. Results from IAL have a slightly higher bias as compared to Pace results, with 54.5% of comparable locations trending higher in total Aroclor PCB concentrations reported from IAL (when the two additional Aroclors are removed). Total PCB by Aroclor analyses seem to be biasing low as compared to congener analysis as 90% of comparable locations have higher total PCB as congener concentration.

The calculated CRM IAL recovery average based on the three CRM PCB in soil samples analyzed was 57.3% with standard deviation of 10.2%, as seen below in the **Figure 2**. The CRM recovery from Congener analysis was 75.2%, and there was a 38% higher CRM recovery for congener vs Aroclor analysis (3.69 vs 2.29 mg/kg, respectively). It is important to note that the all CRM recoveries achieved by IAL and Vista laboratories were within the QC performance acceptance limits identified on the ERA certificates of analysis (CDM Smith, 2018). Both Aroclor and congener recoveries indicate potentially low bias of PCB results. The low bias of IAL's Aroclor analysis compared to congener analysis for CRM samples is consistent with the findings of total PCB concentrations for the split samples, where congener analysis consistently showed higher bias compared to either of the Aroclor analyses (**Figure 1**).

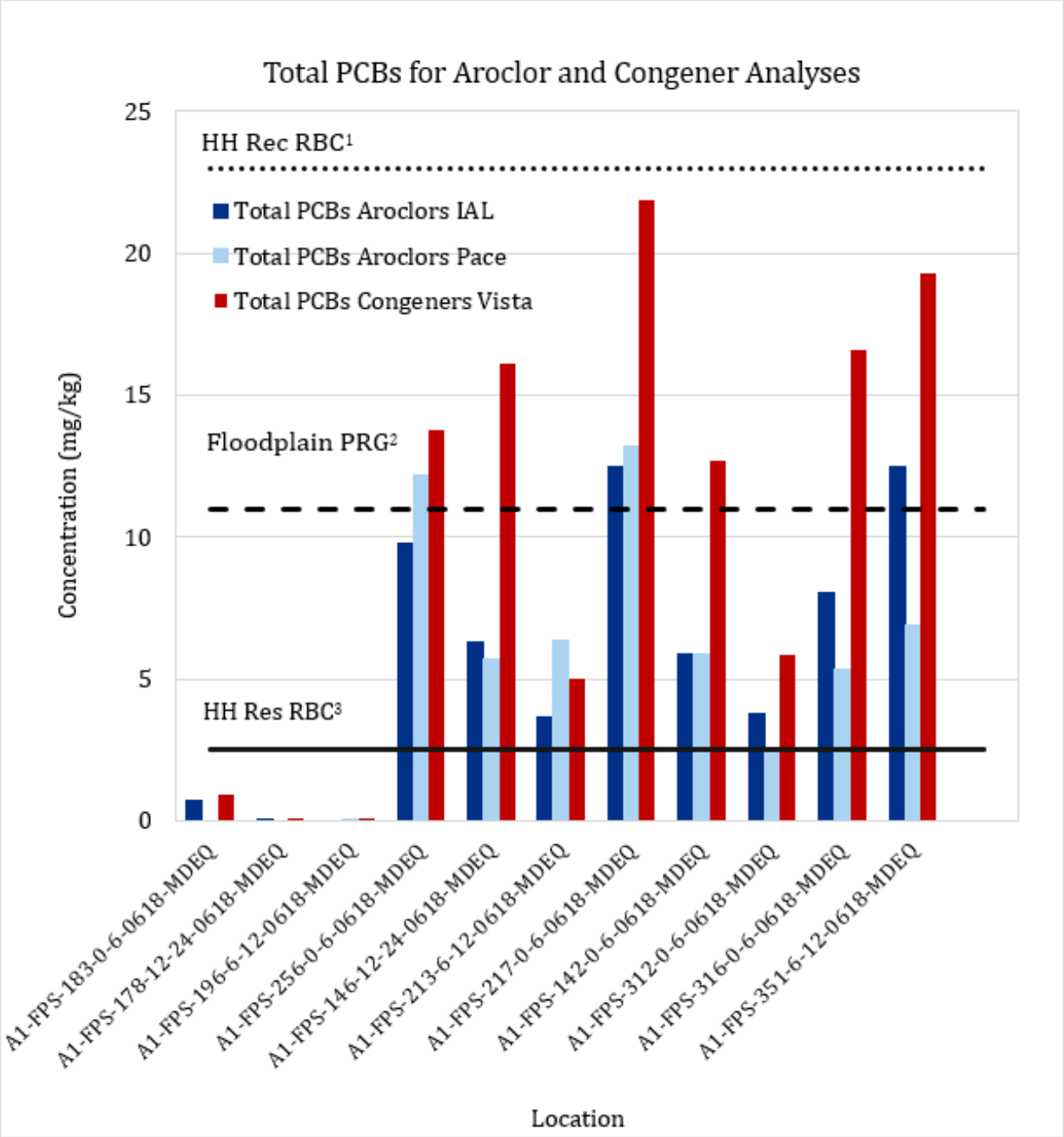

Figure 1 Total PCB concentrations for Aroclor and Congener analyses

[1] Human Health Recreational Risk-Based Concentration
[2] Floodplain Preliminary Remediation Goal
[3] Human Health Residential Risk-Based Concentration

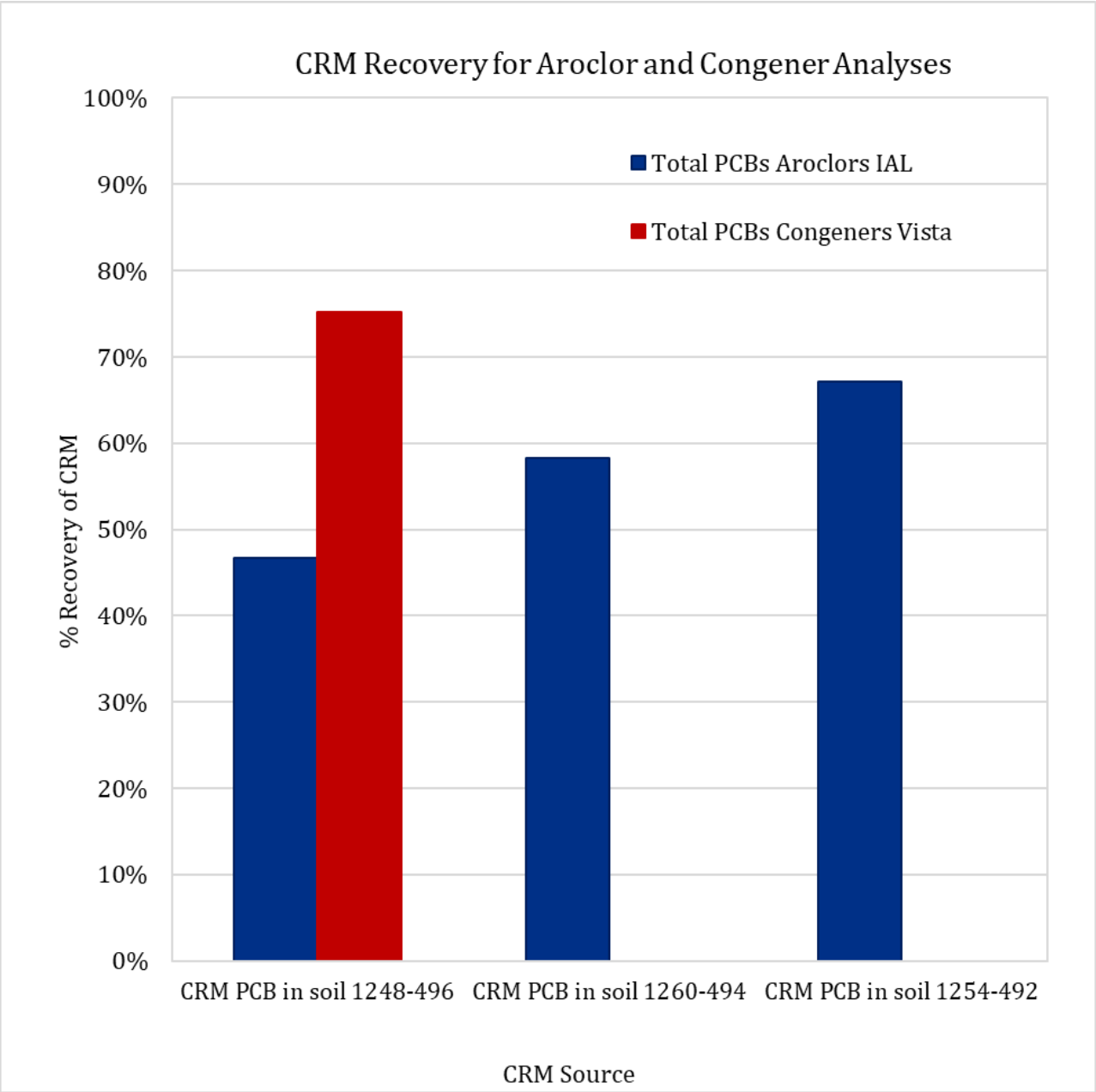

Figure 2 CRM sample recovery for Aroclor and Congener analyses

Conclusions and Recommendations

Based on total Aroclor results from both laboratories and the total congener results received for split sampling for OU-5 Area 1:

- identification and quantitation of individual Aroclors by method 8082A is fairly consistent as performed by each laboratory following their own SOPs for extraction and analysis,
- results are biased low for total Aroclor analysis by method 8082A compared to congener analysis by method 1668A,
- method 1668A congener analysis has a better recovery of the CRM sample, Aroclor 1248, compared to Aroclor analysis by method 8082A,
- the congener results for eight of the eleven split sample sets submitted for congener analysis are greater compared to the total PCB concentration reported by Method 8082A,
- the congener results for four of the eleven split sample sets submitted for congener analysis increased the total PCB concentration over the ecological risk value of 11ppm compared to the lower total PCB concentration reported by Pace.

Based on the CRM results and the total PCB by congener analysis, there appears to be low bias across all soil total Aroclor PCB results, regardless of laboratory. Essentially, Aroclor analyses do not appear to be accurately quantifying the total PCB concentration in site soils. These bias impact not only our understanding of the nature and extent of site contamination, but also the associated potential risks to human health and the environment. Consequently, and based on these split sample results, we recommend proceeding with congener analysis in future sampling efforts. The congener analysis procedure was included as a part of the 2016 QAPP, and it is recommended that the established 2016 QAPP is followed in the future sampling events by utilizing congener analysis for quantification of overall PCB contamination.

There are several ways that congener analysis might be applied at this site that do not include running all samples for congener analysis (the items below are not intended to be an exhaustive list of possibilities):

1. Submit select samples for congener analysis:
    a) Those samples with total Aroclor PCB results approaching within some factor of a site action level.
    b) Other location specific samples of concern based on historical results or other factors.
2. Submit a suitable portion of the samples collected from the site for total PCB by congener analysis to be used in generating site specific correction factors that can be applied to total PCB by Aroclor results.

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